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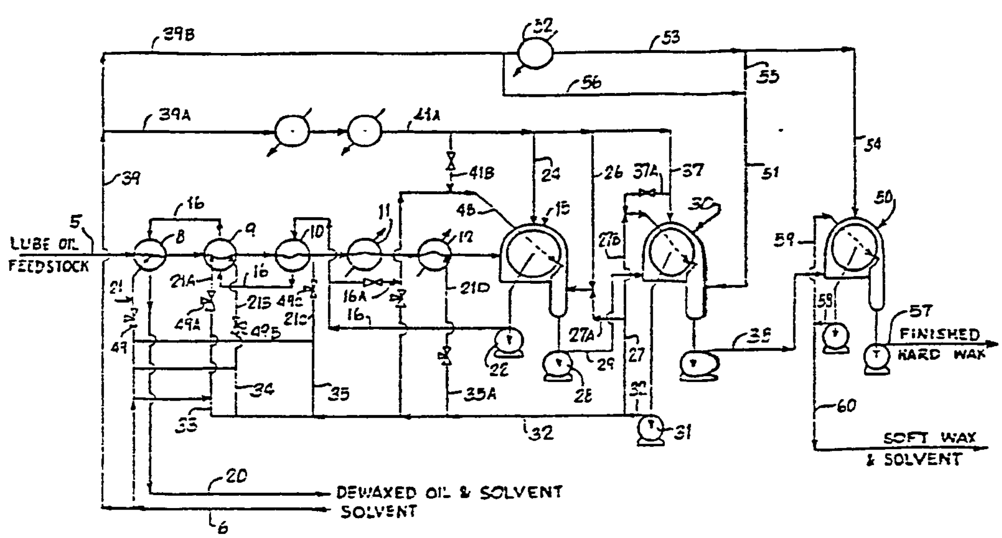



(57) Abstract

A solvent dewaxing process for separation of wax from a waxy petroleum fraction wherein wax crystals are separated from a wax-oil-solvent mixture by filtration at a low temperature in a rotary drum vacuum filter (15) which comprises continuously or periodically backwashing said filter with a relatively large volume of liquid solvent at filtration temperature which improves filtration rates and degree of separation of oil from wax as compared with conventional processes employing "hot washing" of the filter.

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# Solvent Dewaxing Process

This invention relates to a process for solvent dewaxing of waxy petroleum oils and deoiling of petroleum waxes. In one of its more specific aspects, the invention relates to an improved process for the separation of crystalline wax from solvent dewaxed oil by filtration and for maintaining high filtration rates of continuous rotary filters.

A number of methods are known for solvent dewaxing of petroleum oil stocks. In general, a waxy oil stock is mixed with a solvent and the resulting mixture chilled to a temperature at which wax separates from the mixture as solid wax crystals. The wax crystals may be separated from the oil-solvent mixture by filtration and the solvent recovered from the separated wax and dewaxed oil for reuse in the process.

Solvents known to be particularly effective for dewaxing petroleum oils include, for example, methylisobutylketone; mixtures of benzene or toluene and an aliphatic ketone, e.g., acetone, methylethylketone, methylisobutylketone, methyl-n-propylketone; dichloroethane-methylene chloride; and the like. Usually, the preferred aliphatic ketone is methylethylketone or methylisobutylketone and the preferred aromatic hydrocarbon is toluene. Such solvent systems are well

known in the art and are referred to herein simply as solvent or dewaxing solvent.

In most of the industrial processes for the separation of wax from petroleum oil stocks, dewaxing solvent is mixed with a waxy oil stock and the mixture cooled at a controlled rate in a scraped surface heat exchanger. Generally, the oil and part of the solvent are mixed with one another at approximately the same temperature to effect dilution of the oil with solvent before the mixture is cooled or chilled. This mixture of oil and solvent is then cooled, usually with incremental addition of more solvent, at a uniformly slow rate under conditions which avoid vigorous agitation of the solution after the initial formation of wax crystals takes place, i.e. after the cloud point is reached, to permit the growth of relatively large, readily filtrable crystals of solid wax. Such processes are illustrated in U.S. Patents 3,764,517, 4,115,243 and 4,140,620, incorporated herein by reference.

In a typical commercial process, the waxy oil charge is diluted with solvent and heated, if necessary, to a temperature at which all the wax present in the charge is dissolved. The homogeneous charge is then passed to a cooling zone wherein cooling takes place at a uniformly slow rate in the range of about 1 to 10°F per minute (0.5 to 5.6°C per min) until a dewaxing temperature is reached at which a substantial portion of the wax is crystallized, and the dewaxed oil, after separation of solidified wax, has the desired pour point, generally in the range of about 25°C to -40°C). Wax crystals are separated from the mixture of oil and solvent at the dewaxing temperature for recovery of a dewaxed oil-solvent solution and a solid wax containing

a minor proportion of oil (slack wax). The separated oil-solvent solution is further processed for recovery of solvent and product dewaxed oil. The slack wax may be subjected to additional processing for removal of additional oil therefrom.

The recovery of solvent from dewaxed oil and from wax-solvent mixtures produced in solvent dewaxing operations may be effected by distillation. A combination of high and low pressure flash vaporization stages followed by stripping with steam or inert gas is generally preferred. A system for separately recovering two solvents from a dewaxed oil-solvent solution and from a wax slurry by a combination of high and low pressure flash vaporization followed by gas stripping is disclosed in U.S. patent 4,052,294, incorporated herein by reference.

Dewaxing solvents are employed to maintain fluidity of the oil in the coolers and chillers, and may be added before the oil is cooled and in increments during cooling. Often the oil is given a final dilution with solvent at the separation temperature, reducing its viscosity for rapid filtration. Commonly, solvent added to the oil in such processes is at the same temperature, or at a somewhat higher temperature than the oil. Under controlled conditions, wax crystals are formed which are easy to separate from the oil by filtration and which contain little occluded oil.

In commercial solvent dewaxing processes, separation of crystalline wax from dewaxed oil-solvent solutions is commonly accomplished in rotary drum vacuum filters. Rotary drum vacuum filters are common articles of commerce, and are well understood by those skilled in the art.

As conventionally practiced, a mixture of wax crystals, dewaxed oil and solvent flows into the vat of a rotary drum continuous vacuum filter. A segmented rotary drum covered with a filter cloth is rotated so that a segment is immersed in the chilled mixture contained in the filter vat. A wax cake forms on the filter cloth as dewaxed oil and solvent are drawn through the filter cloth under the influence of an imposed vacuum. As each segment of the rotary drum leaves the chilled mixture, excess dewaxed oil and solvent are displaced from the wax cake on that segment of the filter by the flow of inert gas drawn through the cake by vacuum applied to the inner side of the drum. Next the wax cake passes under cold solvent sprays where additional dewaxed oil is displaced from the wax cake by cold solvent drawn therethrough by vacuum. As the wax cake leaves the solvent spray area, inert gas is drawn therethrough once by the imposed vacuum, displacing additional solvent and drying the wax cake. After this second treatment with inert gas, the wax cake passes into a blow-back zone wherein the wax cake is loosened from the filter cloth by a flow of inert gas under pressure from inside the filter drum. The loosened cake is deflected by a deflector blade and falls into a boot, from which the wax is removed for further treatment. After wax removal, the segment of the rotary filter then passes into an area where neither vacuum nor pressure is applied to the filter surface before re-entering the body of chilled mixture for another cycle.

A typical rotary vacuum filter has three separate lines connected to the trunion head through which vacuum is applied to the filter. The bottom connector, commonly called "bottom pick-up" serves the bottom part of the filter drum or that portion immersed in the chilled mix of the wax, solvent and dewaxed oil. The side pick-up

serves the first inert gas flow and the cold solvent spray areas. The top pick-up serves the second inert gas flow area just prior to wax cake discharge.

In commercial dewaxing processes, wax separated from dewaxed oil-solvent mixture in the primary filter is referred to as slack wax, and the filtration step is referred to as primary filtration. This slack wax, in the primary filter boot contains a quantity of dewaxed oil entrained therein. In order to improve recovery of dewaxed oil and improve quality of recovered wax, the slack wax in the primary filter boot may be slurried with additional cold dewaxing solvent for dissolving dewaxed oil, and the slurry separated into a wax cake of substantially reduced oil content and a solvent dewaxed oil solution by a second filter, termed repulp filter. A third stage called wax deoiling is sometimes used. In this stage filtration is conducted at a somewhat elevated temperature to remove essentially all the remaining oil as well as low melting point wax components. Preparation for the deoiling filtration may merely involve dilution with warm solvent (called warm-up deoiling) or it may involve dilution, heating to solution, and chilling with accompanying recrystallization. The process steps in the repulp filters are essentially the same as those described above for the primary filter.

After a period of continuous use, the filter cloths of the continuous rotary filters gradually become partially plugged or "blinded", by the tiny wax particles and/or ice crystals. As a result, the filtration rate of the filters gradually decreases so that it becomes necessary periodically to clean the filter cloths. According to

generally accepted commercial practice, cleaning of filter cloths is presently accomplished by "hot washing" the filter cloths with dewaxing solvent. Such "hot washing" comprises: removing the filter to be cleaned from service and draining chilled mixture from the filter vat; spraying the exterior of the filter cloth on the rotating filter drum with "hot" dewaxing solvent, at a temperature in the range of about 120 to 180° F (59 to 85°C) for a time to dissolve wax and wash it from the filter cloth; draining the washings from the filter vat; rechilling the filter with cold solvent; and, finally, returning the filter to service.

Different dewaxing stocks, or even various lots of the same dewaxing stock, may differ in the rapidity with which they will blind filter cloths. When processing an average filtering stock, the decline in filter rate is gradual and a time schedule is used whereby a particular filter is washed about every six hours. When a stock is processed which blinds the filter more quickly, the hot washing frequency may be increased to maintain a higher average filtration rate.

Backwashing rotary drum vacuum filters with solvent or other fluid from the underside of the filter cloth to the outside, while maintaining the filter in service, is known in processes other than solvent dewaxing of petroleum oils. In the field of solvent dewaxing, it has been proposed heretofore, in U.S. Patent 1,874,972 to continue to apply a gaseous back pressure to the reverse side of the filtering surface after removal of the filter cake and during passage through the filter surface washing zone while simultaneously spraying petroleum naphtha in the form of a finely separated mist against the reverse side of the filtering surfaces into which it is forced by the

gas to remove waxy constituents from the pores to produce substantially clean filter surfaces.

According to the present invention, the filter cloth of a rotary drum vacuum filter is continuously or periodically backwashed with a relatively large volume of cold dewaxing solvent, primary filtrate, or repulp filtrate, effecting removal of solid particles imbedded in the filter cloth and improving filtration rates.

Details of the invention will be evident from the accompanying drawing and the following detailed description of the process of this invention.

The figure is a simplified schematic flow diagram illustrating a solvent dewaxing process in which the primary filters are treated in accordance with the process of this invention.

With reference to the drawing, a waxy petroleum oil feedstock, for example, a wax distillate lubricating oil base stock which has been maintained at a temperature above that required for complete solution of the wax incrementally enters the system through line 5 and is diluted as it is passed through chillers 8, 9, 10, 11, and 12 wherein the mixture is cooled to the desired dewaxing temperature, and delivered to rotary vacuum filter 15.

Suitably, chillers 8, 9, 10, 11, and 12 consist of double wall heat exchangers which comprise an inner pipe through which the solvent-oil mixture is passed, surrounded by an outer pipe or jacket of larger diameter supplied with a suitable coolant or heat exchange fluid. The chillers may comprise scraped wall, double pipe heat exchangers of the type well known in the art.

Coolant, comprising a dewaxed oil and solvent mixture obtained as a cold filtrate from rotary drum vacuum filter 15 as described later, is supplied to the annulus or jacket of chiller 10 through line 16 and thereafter to the jackets of chillers 8 and 9, warming the filtrate and cooling the incoming mixture. The resulting warmed filtrate then flows through line 20 to a solvent recovery system, not shown in the drawing. Chillers 11 and 12 are supplied with coolant from a suitable refrigeration system, not illustrated.

The incoming mixture of oil feedstock and solvent is progressively cooled during its passage through chillers 8, 9, 10, 11, and 12 to a dewaxing temperature which may be in the range of -4°C to -40°C (about 25°F to -40°F). The resulting chilled mixture, comprising dewaxing solvent, oil and wax crystals, is fed to a continuous rotary drum type vacuum filter 15, hereinafter referred to as the primary filter.

As the feedstock passes through the various chillers, solvent may be added incrementally to the mixture, the solvent entering the chiller system from lines 6 and 32, through lines 21, 21A, 21B, 21C, 21D. These dilution solvent rates are controlled with control valves 49, 49A, 49B, 49C, and 49D. Solvent in line 32 is filtrate from the second or repulp stage and contain a minor amount of dewaxed oil. Solvent from lines 6 and 32 may be blended as illustrated for controlling temperature of the dilution solvent. Incremental dilution of waxy petroleum oil stocks, especially lubricating oil base stocks, during the period of chilling and wax crystallization is a technique well known in the art. Typically, the cooling rate in the system of chillers 8, 9, 10, 11, and 12 is within the range of 0.5 to 4.5°C per minute, preferably within the range of 1 to 3°C per

minute. Chillers 11 and 12 suitably comprise scraped wall heat exchangers cooled by a suitable refrigerant, from a source not illustrated in the figure.

It is to be understood that chillers 8, 9, 10, 11, and 12, shown diagrammatically in the figure, may represent groups of heat exchangers, preferably of the double pipe type and typically comprising some 20 to 24 double pipe heat exchangers arranged in four parallel banks and equipped with mechanical scrapers to remove paraffin accumulations from the inner wall of the inner pipe through which the mixture of oil and solvent is passed.

A mixture of dewaxing solvent, dewaxed oil and wax crystals at the desired dewaxing temperature is supplied to primary filter 15, preferably a rotary drum type vacuum filter, wherein the mixture of oil and dewaxing solvent is drawn through the filter and solid wax is retained on the filter surface. Filtrate from the primary filter 15 comprising a mixture of dewaxed oil and dewaxing solvent, is passed by pump 22 through line 16 to the outer pipe or jacket of heat exchanger 10.

Wax cake accumulated on the surface of the filter is washed with cold solvent from line 24, and thereafter removed from the primary filter in a continuous manner, mixed with additional fresh solvent from line 26 or with repulp filtrate from line 27 to form a slurry, and passed by pump 28 through line 29 to repulp filter 30. The repulping filter 30 operates in a manner analagous to that of the primary filter 15 and serves to recover oil retained in the wax cake discharged from primary filter 15. Repulp filtrate from repulping filter 30 is passed by pump 31 through line 32 to lines 33, 34, 35 and 35A for dilution of the lubricating oil feedstock,

and to line 27 for slurrying the wax cake from primary filter 15.

Wax cake accumulating on the filter in repulping filter 30 is washed on the filter with cold clean solvent from line 37 and removed from the filter in a continuous manner. As mentioned earlier, some manufacturers add a third or "wax deoiling" stage. As illustrated, by the "warm-up" procedure, cold wax cake in the wax boot of repulp filter 30 is diluted with warm solvent from line 51 to adjust the temperature of the deoiling mix to the desired value. The deoiling mix then flows by line 38 to deoiling filter 50. The deoiling filter operates in a manner analgous to that of primary filter 15.

From line 6, some of the solvent is passed by way of lines 39 and 39A to chillers 40 and 41, wherein the solvent is cooled to the desired filtration temperature by a suitable refrigerant supplied to the chillers 40 and 41 from a source not illustrated in the drawing. The wash solvent from lines 24 and 37 and solvent from line 26 preferably are chilled to the same temperature as that at which filtration of the dewaxed oil is carried out. The repulping filter 30 operates at essentially the same temperature as that of the primary filter 15.

From line 6, some of the solvent is passed by way of lines 39 and 39B to chiller 52 wherein the solvent is cooled to approximately the temperature at the deoiling filtration. The chilled solvent is then carried by way of line 54 to the wash sprays of deoiling filter 50. Warm-up dilution solvent for the wax leaving repulp filter 30 must be somewhat warmer than the solvent in line 53 so some of the solvent from line 53 flows by

way of line 55, joining a stream of warm solvent from line 39B by way of line 56, thus effecting the desired temperature in line 51 which carries the dilution solvent to the repulp wax cake from repulp filter 30.

As illustrated diagrammatically in the figure, the primary filter 15 is backwashed with cold filtrate from the repulping filter 30. Cold solvent for backwashing the filter is taken from line 32 through line 46 at a rate controlled by control valve 47 and introduced into primary filter 15 through the filter trunion in known manner to that segment of the filter from which wax cake has been removed as illustrated diagrammatically by line 48. The washing is preferably carried out just prior to the beginning of filtration by the application of vacuum to that particular segment of the filter. This location for the backwash is particularly suitable because it does not interfere with normal operation of the filter.

In accordance with the process of this invention, a relatively large volume of cold solvent, suitably filtrate from the repulping filter, is forced under pressure through the filter 15 in reverse flow by pump 31 when valve 47 is opened. The backwash solvent passing through the filter collects in the vat of the filter where it mixes with cold feed supplied to the filter vat from chiller 12, diluting the incoming feedstock. Control valve 49D preferably is used to reduce or discontinue flow of repulping filtrate through line 35A during that period of time when valve 47 is opened. Alternatively, backwash solvent may be cold fresh solvent from line 41A via line 41B or it may be primary filtrate taken from line 16 via line 16A. Repulp filtrate, which comprises a mixture

of solvent and dewaxed oil, is preferred since its use does not impose an additional refrigeration and recovery system load as would be the case for use of fresh solvent.

A suitable alternative backwash location is in the portion of the filter prior to cake discharge not illustrated in the drawing, served by the top pick-up described earlier. To accomplish this, the valve in the top pick-up line must be closed during the backwash interval with the backwash solvent being introduced into the line between this closed valve and the filter trunion head. Either fresh cold solvent from line 41A or repulp filtrate from line 46 are suitable backwash solvents when applied at this location, with backwash solvent going with the wax into the filter boot and serving as repulp dilution.

The pressure applied to the backwash solvent may be varied, depending upon the characteristics of the filter. Pressures in the backwash solvent line in the range of about 12 to 100 psig are generally appropriate. The temperature of the solvent employed as backwash is preferably near the temperature of the mixture undergoing filtration and preferably does not exceed the filtration temperature by more than about 6°C. Higher temperatures may be used, e.g. 10 to 25°C above filtration temperature. If the backwash temperature is too high, a portion of the wax cake may melt blinding the filter cloth, decreasing the filter rate and nullifying some advantages of the process of this invention.

Washing of the filter may be carried out in a continuous or intermittent manner. In one preferred embodiment the washing is carried out intermittently for a period

of 1.5 to 5 minutes at intervals within the range of 20 minutes to two hours. The desired frequency and the length of the wash periods are dependent upon a number of factors including the nature of the charge stock, rates of chilling, rates of agitation during chilling, etc. The rate at which the wash solvent is supplied to the filter should be within the range of about 20 percent to 40 percent of the rated capacity of the filter.

Backwashing of the repulp filter 30 is accomplished by supplying repulp filtrate from line 32 and 27 applied after wax discharge. Fresh cold solvent from line 37 may be supplied as backwash through line 37A. In those cases where a third stage is not used, application of backwash solvent after cake discharge is preferred. When a third stage is used, as illustrated, fresh cold solvent may be preferred as backwash solvent applied before wax discharge; this solvent mixes with the wax and serves as part of the required dilution for the third stage.

Backwashing of the deoiling filter 50 may be suitably carried out as illustrated by use of soft wax filtrate from lines 58 and 59 applied after wax discharge.

Some of the advantages of the method of this invention will be evident from the following example.

Example 1

Tests were made on a commercial continuous rotary drum vacuum filter in a solvent dewaxing operation as described herein.

In these tests, a wax distillate having an API gravity of 34.2°, an initial boiling point of about 600°F; an end point of about 1000°F; a viscosity of about 107.6

SUS at 100°F and 40.5 SUS at 210°F; a pour point of 85°F; and containing about 15 wt. % wax was mixed with about 2.1 volumes dewaxing solvent comprising 35% toluene and 65% methyl ethyl ketone. The wax distillate-solvent solution was chilled, in scraped surface chillers to a temperature of about -10°F, and the cold mixture filtered in a primary continuous rotary vacuum filter as described hereinabove.

The primary rotary vacuum filter was operated according to the conventional method, with periodic washing of the filter cloth with "hot" solvent as described herein, to remove wax particles and ice, after which the filter was again chilled to dewaxing temperature with clean solvent and returned to service.

The "hot washing" procedure is as follows. The supply of chilled feed mixture is cut off from the filter and filtration continued until the level in the filter vat is too low for continued operation. Then, valves in the pick-up connections to the filter are closed so that the filter canvas no longer has vacuum applied from the interior. A drain valve in the filter vat is then opened to drain any remaining chilled mixture from the filter vat. Cold wash solvent of the wash sprays is replaced with hot solvent for at least one cycle to dissolve out wax from the filter canvas. In the absence of vacuum on the interior, these washings flow off the outside of the filter drum through the vat drain to a hot wash receiver. The hot solvent is replaced in the sprays with cold solvent for at least one revolution to rechill the canvas. This solvent also goes to the hot wash receiver. The vat drain is closed and the valve supplying chilled mix to the vat is opened. When level builds up in the vat, the pick-up valves are

opened once again and the filter is returned to service.

In this test example, upon completion of the hot wash, the filter was returned to service, having a filter capacity of about 99%. Filter capacity declined rapidly, and at about the end of the second hour was reduced to about 65% of its rated capacity, where it remained substantially constant for the next four hours. To test the procedure of this invention, the filter was maintained in service and the filter cloth was subjected to a backwash with cold fresh solvent in that portion of the filter just prior to discharge of the wax cake. The cold solvent backwash was applied at a pressure of 18 psig over a filter cloth area described by 72 degrees of arc upon the rotary filter drum, for a period equivalent to two revolutions or about 90 seconds. Filter capacity was restored to full capacity. The filter was maintained in service, without backwash, for a period of six hours, during which time the filter capacity decreased slowly to about 72% of its rated capacity. After six hours of continuous operation the filter cloth was again backwashed for two revolutions with cold solvent, whereupon the filtration rate increased to 96% of its rated capacity.

Thus it is evident that cold solvent backwash in accordance with this invention increases the average capacity of a rotary drum vacuum filter employed in filtering wax crystals from a dewaxed oil-solvent mixture and allows cleaning the filter cloth of accumulated wax and/or resins without taking the filter from service.

In addition to increasing filtration rates and reducing the time period during which a filter must be taken

out of service for "hot washing", the method of this invention unexpectedly produces a wax having a lower oil content than that obtained from operation of a primary filter in the conventional manner as demonstrated in Example 2.

Example 2

In this example, two primary filters were operated simultaneously with the same charge stocks and under the same operating conditions. Filter No. 1 was backwashed in accordance with this invention while Filter No. 2 was operated according to conventional practice by "hot washing" the filter, as indicated in Table I. Two different charge stocks were processed, a waxy distillate and a residuum.

TABLE I

COMPARISON OF OIL CONTENTS OF WAXES
FROM HOT WASHED VS COLD BACK-WASHED FILTERS

| | Filter No. 1 | Filter No. 2 |
|---|---|---|
| Filter Washing Method | Cold Back-Wash[1] | Hot Wash[2] |
| Distillate Charge Stock | | |
| Oil Content (ASTM D721), wt. % | 27.5 | 35[3] |
| Oil Content (ASTM D3235), wt. % | 36[4] | 45.2 |
| Residuum Charge Stock | | |
| Oil Content (ASTM D3235), wt. % | 13.1 | 18.5 |
| Oil Content (ASTM D3235), wt. % | 14.0 | 19.6 |

(1) Cold solvent backwash for approx. 5 min. once per hour
(2) Hot washed at 3 to 4 hour intervals
(3) Estimated by dividing ASTM D3235 value by 1.3
(4) Estimated by multiplying ASTM D721 value by 1.3

It is evident from a comparison of the oil contents of waxes from Filter No. 1 with those from Filter No. 2 that operation of the filters in accordance with the method of this invention produces wax products of improved quality as compared with operation in accordance with conventional hot washing practice.

Claims:

1. In a solvent dewaxing process for separating wax from a waxy petroleum feedstock, wherein a chilled mixture comprising wax crystals, dewaxed oil and a dewaxing solvent is charged to a continuous rotary drum vacuum filter, effecting separation of oil and solvent from said wax and forming a wax cake on said filter followed by washing of said wax cake with solvent and removal of said washed wax cake, the method of improving filtration rates and wax-oil separation which comprises backwashing said filter by forcing a wash liquid comprising said solvent at a temperature not greater than about 50 degrees F above said filtration temperature and a supply pressure in the range of about 12 to 100 psig through said filter in a direction opposite the flow of filtrate therethrough after washing of said wax cake and prior to said vacuum filtration while continuing filtration of said chilled mixture.

2. A method as defined in Claim 1 wherein said back-washing with cold wash liquid is employed for a period of time within the range of 1.5 to 5 minutes at intervals ranging from 10 minutes to six hours.

3. A method as defined in Claim 1 wherein said back-washing follows removal of wax from said filter and precedes vacuum filtration of said chilled mixture.

4. A method as defined in Claim 1 wherein said back-washing accompanies removal of wax from said filter and precedes vacuum filtration of said chilled mixture.

5. A method as defined in Claim 1 wherein the rate of wash liquid employed for backwashing said filter is

within the range of 20 to 40 percent of the rated capacity of said filter.

6. A method as defined in Claim 5 wherein said backwashing of said filter is employed continuously during operation of said filter.

7. A method as defined in Claim 1 wherein said wash liquid temperature is not greater than 10°F above said filtration temperature.

8. A method as defined in Claim 1 wherein said filter is a primary filter and said wash liquid is repulp filtrate.

9. A method as defined in Claim 1 wherein said filter is a repulp filter and said wash liquid is repulp filtrate.

10. A method as defined in Claim 1 wherein said filter is a repulp filter, said wash liquid is clean solvent, and said washing is employed simultaneously with said removal of said wax cake.

11. A method as defined in Claim 1 wherein said filter is a wax deoiling filter and said wash liquid is filtrate from said wax deoiling filter.

12. In a solvent dewaxing process for separating wax from a waxy petroleum feedstock wherein a chilled mixture comprising wax crystals, dewaxed oil and dewaxing solvent is charged to a primary rotary drum vacuum filter at a preselected dewaxing temperature to yield a slack wax cake and a filtrate comprising dewaxed oil and solvent and said slack wax cake is slurried

with additional chilled solvent at said dewaxing temperature for dissolving entrained dewaxed oil therefrom and said slurry is charged to a repulp rotary drum vacuum filter to yield a second wax cake of substantially reduced oil content and repulp filtrate comprising solvent and a minor amount of dewaxed oil, the method of improving filtration rates and reducing the oil content of said slack wax which comprises periodically backwashing said primary filter after the discharge of said slack wax cake with a wash liquid stream comprising said repulp filtrate at a temperature not greater than about 10°F above said dewaxing temperature and at a feed pressure in the range of about 12 to 100 psig while continuing filtration of said chilled mixture.

13. A method as defined in Claim 12 wherein repulp filtrate from said repulp rotary drum vacuum filter is periodically employed as backwashing liquid for said primary filter and for said filter.

14. A method as defined in Claim 12 wherein said second wax cake is slurried with additional clean solvent and said slurry is charged to a deoiling filter at a temperature in the range of 30 to 60°F above said dewaxing temperature wherein a wax product substantially free from oil is recovered as product.

**AMENDED CLAIMS**
(received by the International Bureau on 6 July 1981 (06.07.81))

1. A solvent dewaxing process for separating wax from a waxy petroleum feedstock wherein wax is separated from a chilled mixture comprising wax crystals, dewaxed oil and a dewaxing solvent by filtration with a continuous rotary drum vacuum filter (15) effecting separation of oil and solvent from said wax and forming a wax cake on said filter, and wherein said wax cake is washed with solvent and removed from said filter, characterized by the method of improving filtration rates and wax-oil separation which comprises passing a chilled liquid backwash medium comprising said solvent at a temperature not greater than about 25°C above said filtration temperature through said filter in a direction opposite the flow of filtrate therethrough at a rate within the range of 20 to 40 percent of the total rated capacity of said filter, said backwash following said washing of said wax cake and preceding said vacuum filtration while continuing filtration of said chilled mixture.

2. A method as defined in Claim 1 wherein said chilled liquid backwash is continued for a period of time within the range of 1.5 to 5 minutes at intervals ranging from 10 minutes to six hours.

3. A method as defined in Claim 1 or 2 wherein said backwashing of said filter is employed continuously during operation of said filter.

4. A method as defined in any of Claims 1 to 3 wherein said liquid backwash medium is supplied at a pressure within the range of about 12 to 100 psig.

5. A solvent dewaxing process as defined in any of Claims 1 to 4 wherein wax cake from a primary rotary drum vacuum filter (15) is slurried with a portion of said dewaxing solvent and the resulting slurry is charged to a repulp rotary drum vacuum filter (30) to yield a second wax cake of substantially reduced oil content and repulp filtrate comprising solvent and a minor amount of de-waxed oil, and said liquid backwash medium comprises said repulp filtrate.

## EDITORIAL NOTE

The applicant failed to renumber the amended claims in accordance with Section 205 of the Administrative Instructions.

In the absence of any specific indication from the applicant as to the correspondence between original and amended claims, these claims are published as filed and as amended.

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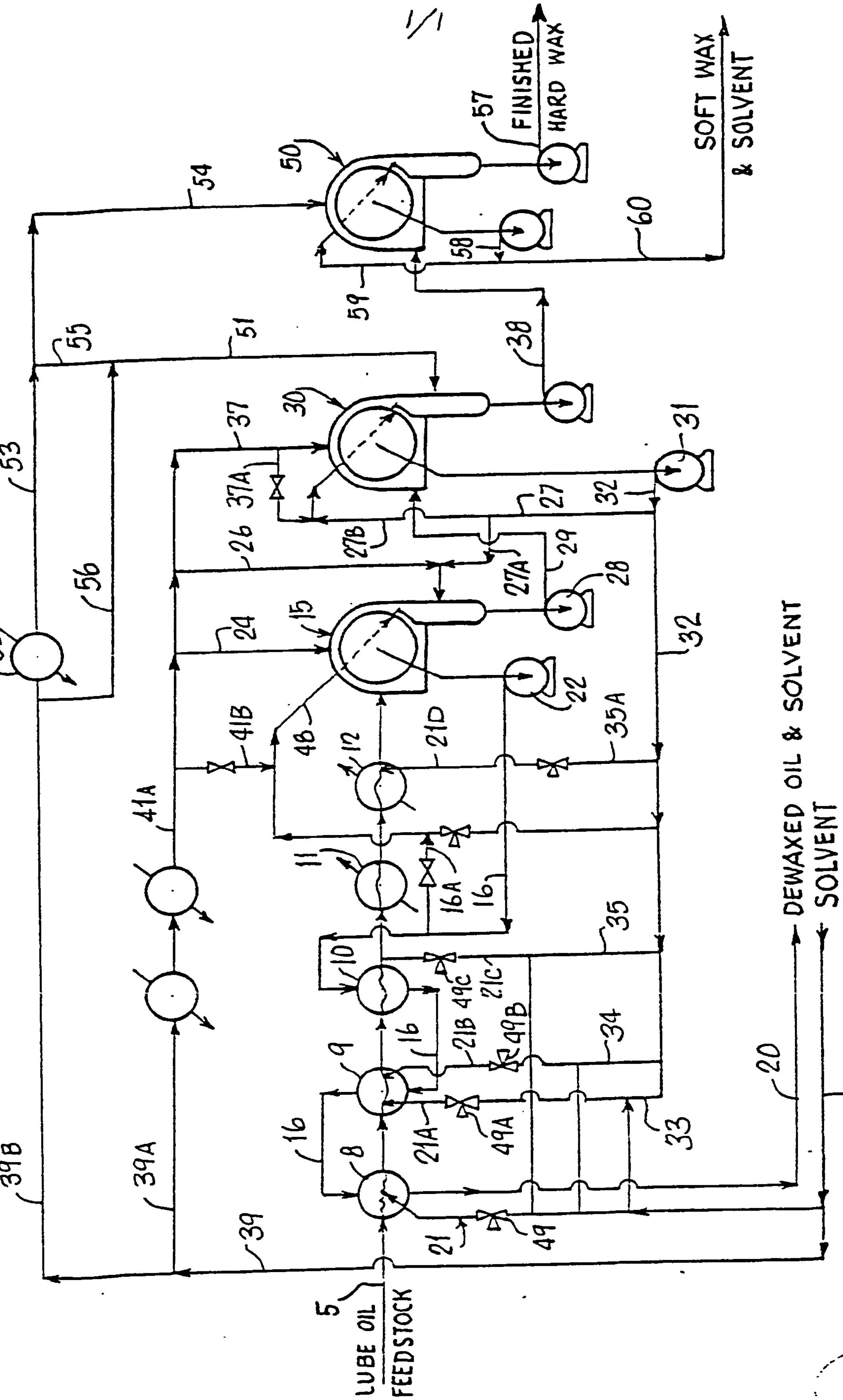
FINISHED HARD WAX
SOFT WAX & SOLVENT
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# INTERNATIONAL SEARCH REPORT

International Application No PCT/US80/00815

**I. CLASSIFICATION OF SUBJECT MATTER** (if several classification symbols apply, indicate all) [3]

According to International Patent Classification (IPC) or to both National Classification and IPC

INT. CL.[3] C01G 43/06
U.S. CL. 208/38; 210/769,772,784,798

**II. FIELDS SEARCHED**

Minimum Documentation Searched [4]

| Classification System | Classification Symbols |
|---|---|
| U.S. | 208/28,38; 210/768,769,772,784,798,805,806 |

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [6]

**III. DOCUMENTS CONSIDERED TO BE RELEVANT** [14]

| Category * | Citation of Document, [16] with indication, where appropriate, of the relevant passages [17] | Relevant to Claim No. [18] |
|---|---|---|
| X | US,A, 1,874,972, PUBLISHED 30 AUGUST 1932, HALL. | 1-14 |
| X | US,A, 3,764,517, PUBLISHED 09 OCTOBER 1973, BODEMULLER, JR. | 1-14 |
| X | US,A, 2,612,466, PUBLISHED 30 SEPTEMBER 1952, KIERSTED, JR. ET AL. | 1-14 |
| X | US,A, 1,857,810, PUBLISHED 10 MAY 1932, GEE. | 1-14 |
| X | US,A, 3,083,154, PUBLISHED 26 MARCH 1963, GERSIC ET AL. | 1-14 |
| X | US,A, 3,093,572, PUBLISHED 11 JUNE 1963, BENEDICT. | 1-14 |
| X | US,A, 3,554,896, PUBLISHED 12 JANUARY 1971, BOZEMAN, JR. ET AL. | 1-14 |
| X | US,A, 4,140,620, PUBLISHED 20 FEBRUARY 1979, PAULETT. | 8-10,12-14 |
| X | US,A, 1,968,239, PUBLISHED 31 JULY 1934, ADAMS. | 8-10,12-14 |
| X | US,A, 2,341,045, PUBLISHED 08 FEBRUARY 1944, KIERSTED, JR. | 4 |

* Special categories of cited documents: [15]

"A" document defining the general state of the art

"E" earlier document but published on or after the international filing date

"L" document cited for special reason other than those referred to in the other categories

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but on or after the priority date claimed

"T" later document published on or after the international filing date or priority date and not in conflict with the application, but cited to understand the principle or theory underlying the invention

"X" document of particular relevance

**IV. CERTIFICATION**

| Date of the Actual Completion of the International Search [2] | Date of Mailing of this International Search Report [2] |
|---|---|
| 19 MAY 1981 | 27 MAY 1981 |
| **International Searching Authority** [1] | **Signature of Authorized Officer** [20] |
| ISA/US | David R. Sadowski<br>DAVID R. SADOWSKI |

Form PCT/ISA/210 (second sheet) (October 1977)